

# 11051 Synthetic Organic Chemicals

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## *Extraction Apparatus for the Laboratory*

THE term extraction denotes the removal of definite constituents of a mixture by selective solution in some liquid in which they alone are soluble. Extraction falls into two classes, involving the treatment respectively of liquids and solids by liquid extraction media. The latter case will first be considered.

The classical method of doing this involves the use of a Soxhlet extraction apparatus, in which the condensed solvent flows onto the mixture of solids contained in a paper extraction thimble of special construction which serves both as container and filter, while the filtered solution is periodically transferred to the boiling-flask by means of an automatic siphon. This ingenious apparatus, in which the extraction is carried out at temperatures below the boiling point of the solvent, suffers from the disadvantage of permitting the extraction of only relatively small quantities, as the capacity of thimbles obtainable in the market is limited.

The extraction of quantities of solids up to about 500 g. may conveniently be carried out without the use of a Soxhlet apparatus merely by placing the substance in a fluted filter paper in an ordinary funnel stoppered to a flask containing the boiling solvent. The wide end of the funnel is loosely closed with a round flask, of larger diameter than the mouth

of the funnel, through which cold water is run (Fig. 1). The vapor of the boiling solvent condenses on the bottom of this flask, and the condensed liquid falls upon the material to be extracted; after percolating through this, it filters through the paper and runs down to the boiling flask. In this way the extraction is carried out at the boiling temperature of the solvent without the use of special apparatus and without particular attention. The water-cooled flask forms a surprisingly efficient condenser, and extractions may be performed even with ether without undue loss of solvent.

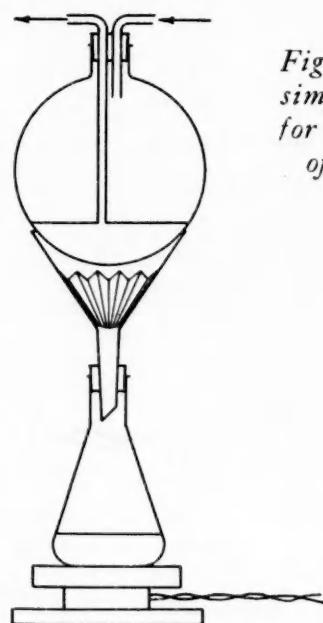


Figure 1 shows a simple apparatus for the extraction of solids up to 500g.

A different type of apparatus must be employed for larger quantities of solids. The material to be extracted may be

treated in a percolator; but, if this is not available, a satisfactory device may be constructed from a bottle of suitable size. This is filled to about three-quarters of its capacity with the solid to be treated, holding a wooden rod in position along the axis of the bottle during the process of filling. This rod is then carefully withdrawn so as to leave a channel, into which a tube is inserted. The apparatus, shown in Fig. 2, is then

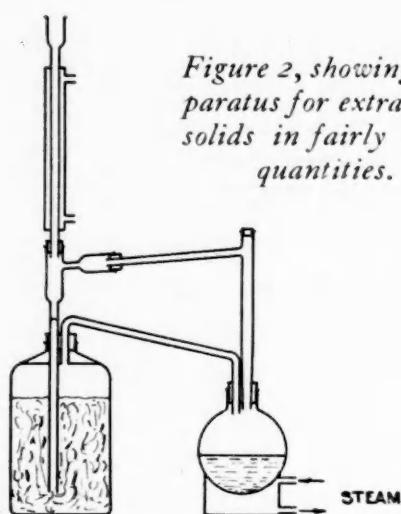


Figure 2, showing apparatus for extracting solids in fairly large quantities.

assembled and the bottle nearly filled with the extracting liquid, which must have a density appreciably lower than that of the solid. A second portion of the liquid is placed in the boiling flask, and gently boiled. The condensed liquid flows to the bottom of the mass and percolates upward, finally escaping from the exit tube and returning to the boiling flask charged with solute. Owing to the difference of the densities of the solid and the liquid, none of the former should rise with the upward stream of liquid to such an extent as to contaminate the outflowing solution. The extraction may be performed at any desired temperature (below the boiling point of the solvent) by surrounding the extraction bottle with a suitable bath. This system is not highly efficient from a thermal standpoint, but has the advantage of requiring very little attention.

Extraction of substances in solution by immiscible solvents is usually effected in the laboratory by shaking in a separating funnel of capacity sufficient to accommodate the entire quantity. When volumes larger than one liter are to be handled, and only two or three changes of solvent are necessary, the shakings may satisfactorily be carried out in bottles; the liquids are separated by siphoning the bulk of the lower layer, decanting the major portion of the upper layer, and completing the separation in a small separating funnel.

When, however, more agitations are necessary to effect the extraction, continuous extraction permits great economy of time to be made. This operation is carried out exactly as in the extraction of larger quantities of solids in a bottle (Fig. 2), the latter being filled to rather more than three-quarters of its capacity with the solution to be treated. Here, again, it is imperative that the solvent should possess a lower specific gravity than the solution. This apparatus is readily adapted to the extraction of amino acids by Dakin's method with butyl alcohol, though it is advisable to modify it by the introduction of an extra condenser, preferably in the form of a water-cooled flask (Fig. 3).

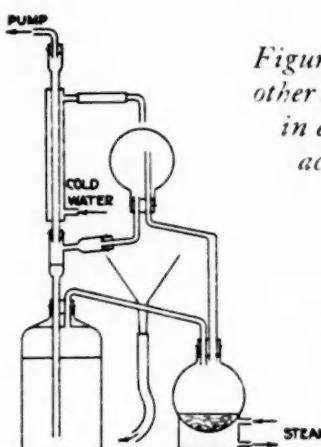


Figure 3, showing another modification useful in extracting amino acids by Dakin's method.

The extraction of solutions by liquids of higher density is less readily adaptable to continuous methods in the laboratory.

## Organic Chemicals as Analytical Reagents

### V REAGENTS FOR IRON

#### THIOGLYCOLIC ACID

Lyons, J. A. C. S. 49, 1916 (1927)

Iron in solutions as dilute as one part in ten million can be detected by this reagent. To 5 cc. of the solution to be examined, which must be free of oxidizing agents, are added one drop of thioglycolic acid and 0.5 cc. of concentrated ammonia. A red or purple coloration shows the presence of iron. The color is due to a complex ferrous ion, any ferric iron present being reduced by the reagent. This reagent in conjunction with the thiocyanate method permits the colorimetric estimation of both ferrous and ferric iron in the same solution.

#### ALLOXANTIN

Deniges, C. R. 180, 519 (1925)

A blue color is produced by this reagent with iron salts in alkaline solution. The presence of citrates or tartrates which give trouble with the usual reagents for iron causes no difficulty in this case. The reagent is prepared by dissolving 0.1 gm. of alloxantin in 10 cc. of normal sodium hydroxide. Any color at this point should be destroyed by boiling and then cooling rapidly. Two cc. of a solution containing one milligram of iron per liter gives a distinct blue color with 1 cc. of the reagent.

#### DINITRORESORCINOL

Nichols and Cooper, J. A. C. S. 47, 1268 (1925)

This reagent in neutral solution gives an olive-green color with iron, brown with copper, and orange-brown with cobalt. The limits of detection in milligrams per cc. are 0.0035, 0.0045, and 0.0033 in the order named. The reagent is prepared by adding an excess of the compound to boiling water and then filtering. Addition of a little sodium acetate increases the sensitivity to iron.

#### DIPHENYLAMINE

Knop, J. A. C. S. 46, 263 (1924)

One gram of diphenylamine in 100 cc. of concentrated sulfuric acid forms a suitable indicator in titrating ferrous salts with tenth-normal potassium dichromate. A mixture of sulfuric and phosphoric acids, also required, is made by mixing 150 cc. of sulfuric acid (s.g. 1.84) with 150 cc. of phosphoric acid (s.g. 1.7) and diluting with water to 1,000 cc. The ferrous solution is acidified with 20 cc. of 1:1 hydrochloric acid, 1.5 cc. of the phosphoric acid mixture, and 3 drops of the indicator. After diluting to 150-200 cc. the solution is titrated with the dichromate. The endpoint is taken as the last drop, causing an intense blue-violet coloration which remains unchanged after further addition of dichromate. A correction of 0.05 cc. is subtracted from the volume of dichromate to account for that used in oxidizing the diphenylamine.

## New Chemicals

The following preparations have been added to our list since the last issue of this bulletin:

- \**n*-Amyl Disulfide
- Amylene (Tech.)
- \**n*-Amyl Iodide
- \**n*-Amyl Mercaptan
- \**n*-Amyl Sulfide
- \* $\beta$ -Bromopentane
- \*p-Bromophenacyl Bromide
- \**n*-Butyl Oleate
- \*Di-*n*-Amylamine
- Ethyl "Oenanthane" (Tech.)
- Hexaethylbenzene
- \*p-Nitrosophenol Sodium Salt
- \*Pentane from Petroleum (Pract.)
- o-Phenetidine (Pract.)
- Phosphorus Pentasulfide (Pract.)
- \*Tri-*n*-amylamine

\*Made or purified in the Kodak Research Laboratories.

## A List of Inexpensive Organic Chemicals

At the suggestion of the Division of Chemistry and Chemical Technology of the National Research Council, we have prepared the following list of inexpensive organic chemicals. They are listed roughly in the order of their prices.

We shall gladly refer inquiries for large amounts to the manufacturers.

Benzene	Glycerol	Benzoic Acid
Naphthalene	Benzyl Chloride	Cyclohexanol
Cymene (Spruce Turpentine)	$\alpha$ -Chloronaphthalene	Ethylene Bromide
Dextrose (Glucose)	Ethylene Chlorohydrin (40%)	Ethyl Phthalate
Toluene	Paraldehyde	Hexachloroethane
Carbon Tetrachloride	Butyl Alcohol	<i>dl</i> -Malic Acid
Calcium Cyanamide	<i>p</i> -Dibromobenzene	<i>m</i> -Nitroaniline
Chlorobenzene	2,4-Dinitrophenol	<i>p</i> -Nitrotoluene
Nitrobenzene	Tetrachloroethylene	Safrole
<i>o</i> -Nitrotoluene	Acetanilide	Sodium Naphthionate
Sodium <i>p</i> -Toluenesulfonate	Acetic Anhydride	Tricresyl Phosphate
Xylene	Calcium <i>l</i> -Malate (Sugar Sand)	Acetyl Salicylic Acid
Ethylene Chloride	Decahydronaphthalene	Anthracene (80-85%)
<i>o</i> -Dichlorobenzene	Diacetone Alcohol	Benzaldehyde
Ethyl Acetate	Dimethylaniline	Benzidine
Formic Acid	Methyl Ethyl Ketone	Butyl Phthalate
Methyl Acetate	Methyl Sulfate	Camphor
Methyl Alcohol	$\beta$ -Naphthol	Chloral Hydrate
Oxalic Acid	$\alpha$ -Naphthylamine	<i>o</i> -Chlorobenzoic Acid
Trichloroethylene	$\alpha$ -Nitronaphthalene	<i>o</i> -Chlorophenol
Formaldehyde (35-40%)	Phthalic Anhydride	Cyclohexane
Tetrachloroethane	Propionic Acid	2,5-Dichloronitrobenzene
Ethyl Propionate	Salicylic Acid	2,4-Dichlorophenol
Acetic Acid	Trichlorobenzene	Diethylaniline
Acetone	Ethylene Glycol	Diphenylguanidine
Sodium Sulfanilate	Methyl Salicylate	Ethyl Butyrate
<i>o</i> -Toluidine	Pentachloroethane	Ethylene Glycol Monobutyl Ether
Cresol (m and p mixture)	2,4-Dinitrochlorobenzene	Ethyl Lactate
Acetyl <i>o</i> -Toluidine	Diphenylamine	Ethyl Oxalate
Aniline	Terpineol	Ethyl <i>p</i> -Toluenesulfonate
Dichloroethylene	Acetonitrile	Gallic Acid
<i>m</i> -Dinitrobenzene	Aldol	Lactic Acid
Dinitrotoluene	Amyl Acetate	Methyl Benzoate
Lactose (Milk Sugar)	Tert.-Amyl Alcohol	$\beta$ -Naphthylamine
Potassium Ethylxanthate	Amyl Phthalate	<i>p</i> -Nitroaniline
<i>p</i> -Dichlorobenzene	Betaine Hydrochloride	Nitrobenzaldehyde (o and m mixture)
<i>p</i> -Toluenesulfochloride	Butyl Propionate	<i>o</i> -Nitrophenol
Oleic Acid	Carbazole	<i>p</i> -Nitrophenol
Sodium Naphthalene- $\beta$ -sulfonate	Citric Acid	Phenyl Salicylate
Benzotrichloride	<i>p,p'</i> -Dichloroazoxybenzene	Quercitrin (Lemon Flavine)
Butyl Acetate	Ethyl Bromide	Sodium Picramate (Paste)
Chloroacetic Acid	Indophenol (Paste)	Sulfanilic Acid
<i>o</i> -Cresol	Mucic Acid	Tannic Acid
$\beta,\beta'$ -Dihydroxyethyl Ether	<i>p</i> -Nitroacetanilide	Triphenylguanidine
Maleic Acid	<i>o</i> -Nitrochlorobenzene	Diphenyl Ether
Stearic Acid	Iso-Propyl Alcohol	Cineole
Urea	Pyridine	Ethyl Formate
Tetrahydronaphthalene	Sodium 2-Chlorotoluene-4-sulfonate	Furfuramide
Chloroform	Sodium Formaldehyde Sulfoxylate	Hexachlorobenzene
Dinitronaphthalene	Sodium <i>p</i> -Phenolsulfonate	Hexamethylenetetramine
Ethyl Carbonate	Terpin Hydrate	<i>p</i> -Nitrophenetole
<i>p</i> -Nitrochlorobenzene	<i>p</i> -Toluenesulfonamide	<i>m</i> -Phenylenediamine
Phenol	<i>p</i> -Toluenesulfonic Acid	Sodium Naphthalenedisulfonate
Tartaric Acid	Paraformaldehyde (Trioxy-methylene)	Tetrachlorobenzene
Thiocarbanilide	Triphenyl Phosphate	Triacetin
Ethylene Glycol Monoethyl Ether	Xyldine	
Furfural		